

Paper No. 14

DIRECT VAPOR CHROMATOGRAPHIC  
DETERMINATION OF MENTHOL, PRO-  
PYLENE GLYCOL, NICOTINE, AND  
TRIACETIN IN CIGARETTE SMOKE.

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ABSTRACT

A simple method which requires no separation techniques is presented for the determination of menthol, propylene glycol, nicotine, and triacetin in cigarette smoke. The procedure consists of smoking onto a Cambridge filter which quantitatively collects the particulate phase, and the smoke components are then distilled directly from the filter in a stream of helium onto a chromatographic column for analysis. All four components are determined from a single chromatogram.

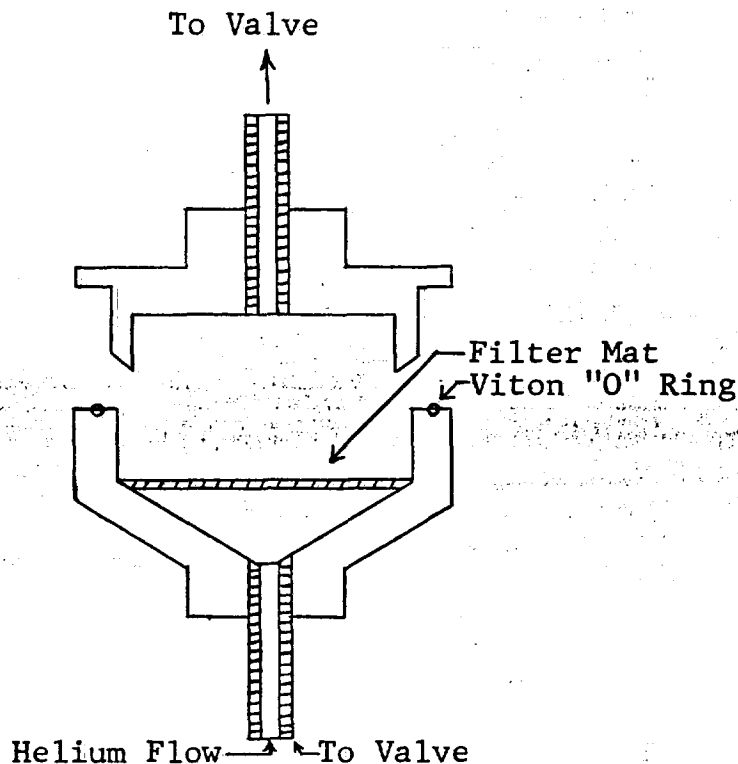
REVIEW BY R. D. HELLAMS

The gas chromatographic method presented by Mr. Lyerly for the determination of menthol, propylene glycol, nicotine, and triacetin in cigarette smoke utilizes a Cambridge filter for the collection and introduction of the smoke onto the gas chromatographic column.

The particulate phase from a single cigarette is collected on a Cambridge filter using the standard puff of 35 cc. for 2 seconds each minute. A modified Cambridge filter holder (Figure 1) used to hold the Cambridge filter is then connected to a heated sampling valve connected to the injector of the Barber-Coleman 5000 Selecta System gas chromatographic instrument which utilizes a thermal conductivity cell detector. The opposite end of the special Cambridge filter holder is connected to a valve which controls helium flow through the Cambridge filter holder.

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FIGURE 1



A fluidized sand bath at 200°C is used to heat the filter holder while the menthol, propylene glycol, nicotine, and triacetin are swept onto and collected in the initial part of the 6' x  $\frac{1}{4}$ " column (14% DEGS on Chromosorb W) at 50°C. After a ten minute sweep period the sampling valve is changed to reroute the flow.

The column temperature is programmed from 50°C to 175°C at 12°/minute for ten minutes, after which the column remains at 175°C until the final peak, triacetin, is eluted and the analysis is completed in 22 minutes.

A calibration curve was prepared by adding the four components in chloroform to a Cambridge filter with a microliter syringe and analyzing the material on this filter by the same procedure as a smoke sample.

The analysis of menthol in cigarette smoke for four brands is given in Table 1, while a typical puff-by-puff analysis of menthol in cigarette smoke is given in Table 2.

TABLE 1

Analysis of Menthol in Cigarette Smoke

<u>Brand</u>	<u>Mg. Menthol/Cigarette</u>
A	0.39
	0.43
	0.40
B	0.48
	0.49
	0.50
C	0.30
	0.29
	0.31
D	0.23
	0.21
	0.22

TABLE 2

A Typical Puff-by-Puff Analysis of Menthol in Cigarette Smoke

<u>Puff No.</u>	<u>Mg. Menthol/Puff</u>
1	0.030
2	0.031
3	0.036
4	0.036
5	0.038
6	0.045
7	0.045
8	0.045
9	0.042

Typical results for the analysis of propylene glycol, nicotine, and triacetin in cigarette smoke by the above presented gas chromatographic method are given in Table 3.

TABLE 3

Analysis of Propylene Glycol, Nicotine, and Triacetin in Cigarette Smoke by Vapor Chromatography

<u>Sample No.</u>	<u>Propylene Glycol mg./cigt.</u>	<u>Nicotine mg./cigt.</u>	<u>Triacetin mg./cigt.</u>
A	0.44	1.23	0.31
	0.42	1.21	0.32
	0.39	1.38	0.32
	0.38	1.21	0.32
B	1.01	1.45	0.25
	1.03	1.50	0.25
	0.98	1.45	0.31
	0.95	1.41	0.33

Table 4 gives a comparison of this gas chromatographic method with other methods for the analysis of propylene glycol and nicotine in smoke.

TABLE 4

Comparison of Vapor Chromatographic Analysis of Propylene Glycol and Nicotine in Smoke with Other Methods

<u>Brand</u>	<u>Propylene Glycol, mg./cigt.</u>		<u>Nicotine, mg./cigt.</u>	
	<u>VPC</u>	<u>Colorimetric</u>	<u>VPC</u>	<u>Spectrophotometric</u>
A	0.38	0.28	1.16	1.05
B	0.95	0.62	1.06	1.11
C	0.96	0.85	1.31	1.31
D	0.34	0.23	0.75	0.76

The low results for the propylene glycol by the colorimetric method were explained by the speaker as being due to the fact that in the colorimetric method the propylene glycol is not separated from the other smoke components as it is in the gas chromatographic method.

The transfer of propylene glycol and nicotine from the Cambridge filter to the gas chromatographic column versus the direct injection of these standards onto the gas chromatographic column was checked. The same peak heights occurred in the two cases.

The speaker summarized his presentation by saying that in this gas chromatographic method no chemical separation is required, quantitative data for four compounds may be made on a single cigarette, total sample from a single cigarette is used instead of aliquots, and a puff-by-puff analysis is possible for any component or for all four components.

Questions asked by the audience after the speaker's presentation of his paper are as follows:

1. Is the Cambridge filter treated? Answer: No.
2. Why did water not show on the chromatogram?  
Answer: The water was removed from the column during the 10 minute sweeping period before programming the column temperature.
3. Were these experiments done to check the quantitative collection of these four compounds on the Cambridge pad during smoking? Answer: No.
4. Was there any study done with the gas chromatographic method for the analysis of smoke where any of the four compounds was not present? Answer: No.